X-ray study of low-temperature annealed arsenic-implanted silicon

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Low-temperature anneals (500–650 °C) of 2, 4, and 8×10^{15} cm^{-2} As^+ implanted in ⟨100⟩ silicon at 50 keV were studied by x-ray double crystal diffraction. The rocking curves were analyzed by a kinematical model. Two regions of strain were found in the solid-phase epitaxially regrown layer. One layer was uniform and positively strained. The other was nonuniform and negatively strained. By comparing rocking curves of repeatedly etched layers it was found that the surface layer is negatively strained, corresponding largely to the substitutional As in the regrown layer. The positively strained region lies at the interface between the implanted layer and the undamaged silicon substrate.

I. INTRODUCTION

Low-temperature anneals in the range 500–650 °C of amorphized As^+ source/drain implants have been used prior to high-temperature anneals at 900–1050 °C to achieve better control of junction depths in shallow-junction NMOS devices. Annealing of the surface-implanted layer occurs through solid-phase epitaxial regrowth (SPE). Growth rates have been found to be dependent on orientation and dopant.[1–4] For moderate-dose As^+ implants, low-temperature annealing was found to proceed with near 100% activation for ⟨100⟩ and substantially lower activation for ⟨111⟩ orientations.[5] Evidence has been found for substitution of high-dose arsenic implants in excess of the solid solubility to approximately 9×10^{21} cm^{-3}.[6,7] However, the apparent solid solubility limit found by RBS is substantially higher than the electrically active fraction in high-dose low-temperature-annealed As^+ implanted in ⟨100⟩ silicon.[8] Short laser anneals of high-dose implants followed by thermal anneals in the range 400–900 °C indicated a limiting solid solubility of 3×10^{20} cm^{-3} for ⟨100⟩ silicon.[9]

We have investigated annealed implants by double-crystal diffraction. The advantage of this method lies in the sensitivity of the resulting rocking curves to strain in the layer. Rocking curves have been used elsewhere to determine the strain of thicker, implanted,[10,11] and annealed[12–14] layers. Following the approach of Ref. 10 we have interpreted our rocking curves using kinematical diffraction for the thin (<0.1 μm) surface layers and perfect crystal dynamical theory for the substrate contribution.

II. EXPERIMENT

100-mm-diam, ⟨100⟩-oriented, 14–20-Ω cm boron-doped silicon wafers were implanted with As^+ at 2, 4, and 8×10^{15} cm^{-2} at 50 keV in a Lintott IIIA implanter. The misalignment direction was 7°, with random misorientation direction. The wafers were then annealed at 500 to 650 °C for 120 min in an N_2 ambient. The resistivity was measured with an in-line automatic four-point probe. Rocking curves about the Si (4 0 0) reflection were measured in the + − mode on a double-crystal diffractometer built at Burroughs using Cr$K\alpha$ radiation, where $\lambda = 2.29$ Å. The line focus was used with the tube operating at 1600 W. The beam was collimated with a broad slit (1×5 mm). The diffracted beam was measured with an open detector.

To demonstrate the change on the rocking curve of fully annealed arsenic-implanted silicon, one wafer implanted at 8×10^{15} cm^{-2} at 50 keV was annealed at 950 °C for 45 min. The rocking curve for this sample is superimposed on a rocking curve for a virgin silicon wafer in Fig. 1. The rocking curve of the implanted and annealed wafer exhibits broadening on the $+\Delta\theta$ side of $\Delta\theta = 0$. $\Delta\theta = 0$ corresponds to the peak of the bulk substrate maximum. This angle for the (4 0 0) reflection of Si is 57.5° for Cr$K\alpha$ radiation. Broadening of the rocking curve on the $+\Delta\theta$ side of $\Delta\theta = 0$ corresponds to a contraction of the lattice due to substitutional arsenic.

Rocking curves for 2, 4, and 8×10^{15} cm^{-2} As^+ implants at 50 keV are shown in Fig. 2. The 8×10^{15} cm^{-2} annealed implants exhibit more structure than the 2 or 4×10^{15} cm^{-2} implants. Two resolved or partially resolved maxima can be observed for the 550–650 °C anneals. One of the maxima lies on the $+\Delta\theta$ side of $\Delta\theta = 0$. The other lies on the $-\Delta\theta$ side. In addition, the secondary maxima on the $+\Delta\theta$ side are more broadened than the maxima on the $-\Delta\theta$ side. For the 2 and 4×10^{15} cm^{-2} implants this broadening is also apparent. The rocking curve of the 8×10^{15} cm^{-2} implant annealed at 500 °C exhibits lower intensity and broadening of the primary and secondary maxima on both sides of $\Delta\theta = 0$.

Profiles of the As distribution for 2, 4, and 8×10^{15} cm^{-2} at 50 keV were calculated by SUPREM III[15] and are shown in Fig. 3. Since we know from Fig. 1. that As contracts the silicon lattice we would expect the maximum surface tensile strain at approximately 400 Å from the surface, with the strain falling off rapidly with depth. A simple model assuming substitutional tensile strain only does not fit the data. The rocking curves for all the implant conditions are best fit by a model containing two regions of strain. One

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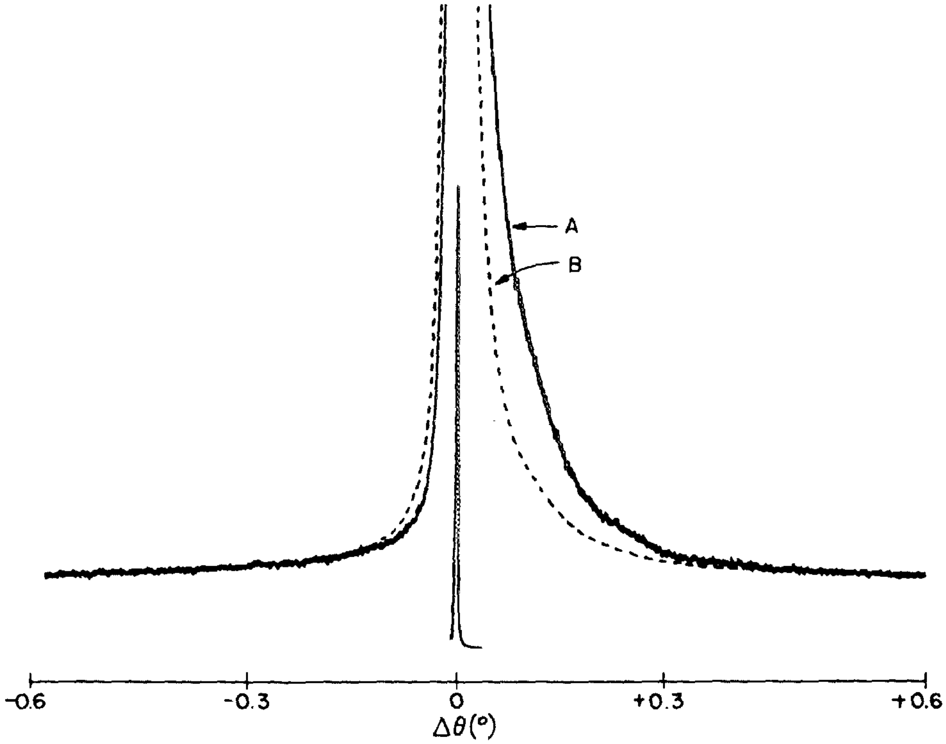

FIG. 1. Rocking curves of (A): an 8×10^{15} cm^{-2} As implant annealed at 950 °C for 45 min; (B) virgin silicon wafer. The attenuation of the bulk silicon peak at $\Delta\theta=0$ is 5×10^{2}.

region is positively strained, implying lattice expansion; the other is negatively strained. X-ray absorption for these layers is quite weak and cannot be utilized to distinguish the ordering of the strain within the layer. However, it was felt that we could determine the ordering of the positively and negatively strained regions by repeatedly etching the surface and fitting the resulting rocking curves to a kinematical model.[10]

To determine the order of strain in the layer, an 8×10^{15} cm^{-2} As^{+}-implanted wafer was annealed at 550 °C and repeatedly argon sputter etched to remove silicon from the surface. Figure 4 (exp) represents experimental data for the unetched sample and rocking curves with 100, 200, and 400 Å removed by argon sputter etching. It was separately determined that argon sputter etching under the same conditions induced no damage observable in the rocking curves for virgin silicon wafers.

Even though the strain through the layer is most likely

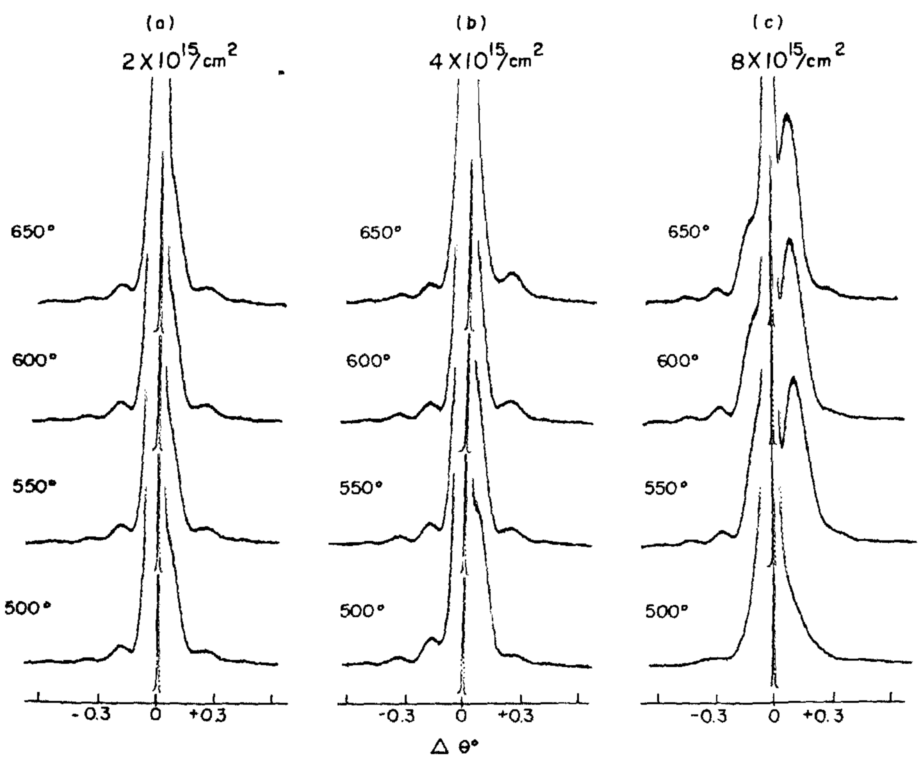

FIG. 2. Rocking curves for 2, 4, and 8×10^{15} cm^{-1} As^{+} implants annealed from 500 to 650 °C. Attenuation of the bulk silicon peak at $\Delta\theta=0$ is 5×10^{2}.

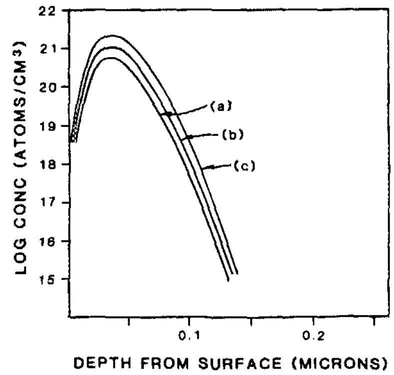

FIG. 3. Calculated arsenic concentrations as a function of depth for (a) 2×10^{15} cm^{-2} (b) 4×10^{15} cm^{-2}, and (c) 8×10^{15} cm^{-2} all at 50 keV.

smooth and nonuniform, a five-lamina model was adequate to fit these data. The ordering of the laminas which best fit the data for the etched layer is a negatively strained surface layer and a positively strained region at the initial interface between the damaged layer and the undamaged silicon substrate [see Fig. 4, Calc. (2)]. Reversing the order of the strain, i.e., with the positively strained region at the surface, did not fit the data nearly as well (see positively Fig. 4, Calc. 1).

The strain models for both the surface-positive and the surface-negative strain are shown at the top of the calculated rocking curves for each of those conditions in Fig. 4. The calculated strain through the layer varies between $+0.15\%$ and -0.25% as indicated in the calculated profiles in Fig. 4. Varying the strain of any of the laminas by more than 10% produced a worse fit to the experimental data. The total thickness of the strained layer is 950 ± 50 Å as calculated from Eq. (6) in Ref. 10. We found that the calculated strain profiles for the 600 and 650 °C anneals were quite similar to the strain profiles calculated for the 550 °C anneal. The strain profiles for the 2 and 4×10^{15} cm^{-2} implants could be fit using different strain profiles. We could fit the data for these implants using uniform two-layer strain distributions, one positive and one negative. This suggests that the double-crystal method is not as sensitive for these doses.

The resistivity of the implants increases with the anneal temperature for all implant conditions except for 8×10^{15} cm^{-2} at 500 °C. This is shown in Fig. 5 and suggests that the activated fraction decreases with increasing temperature. The 8×10^{15} cm^{-2} dose annealed at 500 °C is a special case as indicated by the very-high sheet resistance as well as the thin regrown region indicated in the rocking curve (Figs. 2 and 4). The thickness of the regrown layer for this sample is calculated from the rocking curve as 650 Å. A reasonable scenario

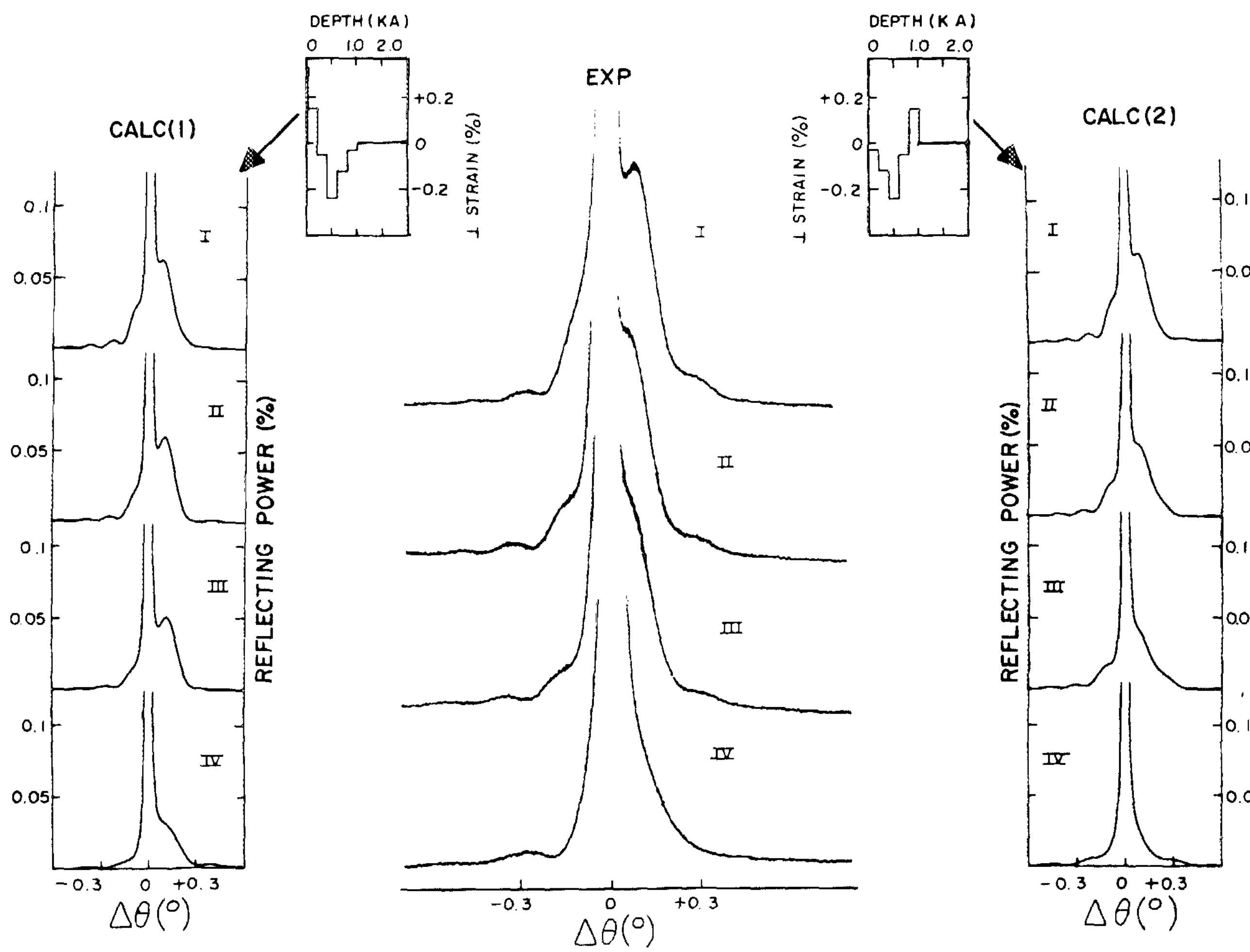

FIG. 4. Theoretical and experimental rocking curves for a sequentially etched layer of As implanted at 8×10^{15} cm^{-2} and annealed at 550 °C. Calc. 1. Theoretical fit assuming (+) strained sublayer at the surface. Exp. Experimental data. Calc. 2. Theoretical fit assuming (−) strained sublayer at the surface. For all conditions: I is unetched, II refers to 100 Å etched from the surface, III refers to 200 Å etched from the surface, and IV refers to 400 Å etched from the surface.

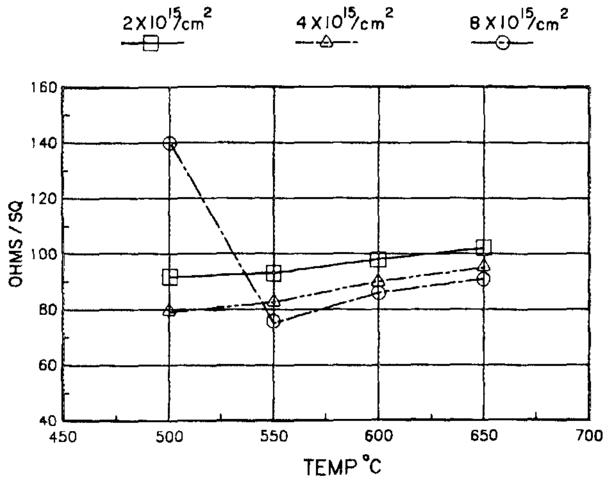

FIG. 5. Resistivity data for 2, 4, and 8×10^{15} cm^{-2} As$^+$ implants annealed in the range 500–650 °C.

for the growth mechanism for this case would be solid-phase epitaxial growth from the interface up to the region of maximum concentration. In this region the high concentration of dopant retards the growth rate sufficiently so as to allow polycrystalline silicon precipitation to dominate the anneal process. This film has a slightly pink hue after anneal, characteristic of doped polycrystalline silicon. Polycrystalline silicon precipitation has been observed by others during anneal, especially for low-solubility species or for very-high-dose soluble implants.[3,7]

III. DISCUSSION

It has been suggested that during these low-temperature anneals the implanted layer initially grows with nearly 100% substitution. This is followed by the formation of arsenic precipitates which decrease the active dopant fraction.[9,16] If the layer were to grow epitaxially with nearly 100% substitutional As, it would be negatively strained. This is observed in the rocking curves. However, there may be defects present in the regrown layer following the 120-min anneal. The positively strained region found in the rocking curves is most likely due to atoms in interstitial sites, either the unannealed implanted As atoms or silicon dislodged from normal sites. These atomic species lie at the interface between the initial implant damaged layer and the undamaged silicon substrate. This is consistent with TEM data which show residual interstitial dislocation loops at the interface.[6] In addition, a theoretical treatment of secondary defects in silicon caused by implanting ions below a critical dose, suggests that interstitials are generated following anneals in the range of 350–650 °C. The critical dose is the level required for amorphizing single-crystal silicon.[17]

A significant feature of these rocking curves is the shift of the primary and secondary maxima toward $-\Delta\theta$ as the temperature is increased. This implies a lower substitutional fraction with increasing temperature and is consistent with the resistivity data and RBS results reported elsewhere.[8]

The retardation of growth rates, especially in the region about R_p, or maximum dopant concentration, associated with very-high-dose implants has been attributed to interfacial strain.[7] We have observed that the strain for the very-high-dose case is not excessive, i.e., is less than 0.25% (see the calculated strain profiles in Fig. 4), and is far less than that observed for boron-implanted and annealed silicon.[13]

IV. CONCLUSION

We have demonstrated the use of double crystal diffraction in the analysis of very thin SPE layers in silicon. Two sublayers have been found for high-dose As$^+$ implants followed by low-temperature anneals in the range 500–650 °C. One of these layers is negatively strained indicating arsenic substitution. The other layer is positively strained. To determine the location of the positively and negatively strained regions, an 8×10^{15} cm^{-2} As$^+$ implant annealed at 550 °C was etched repeatedly until 400 Å was removed. Rocking curves of these etched layers were fit by a kinematical model.[10] The best fit for these layers is a region of negative strain at the surface followed by a narrower region of positive strain at the interface between the implant-damaged surface and the undamaged underlying silicon substrate. These data support a mechanism for the solid-phase epitaxial regrowth which incorporates substitutional As, with residual interstitial As and/or Si atoms remaining at the interface between the annealed layer and the underlying undamaged silicon substrate.

We have found a shift toward $-\Delta\theta$ with increasing temperature for the primary and secondary maxima, most notably for the 4 and 8×10^{15} cm^{-2} conditions. We have interpreted this as a reduction in substitutional As with increasing anneal temperature in the range 500–650 °C. This is consistent with the increase in resistivity with temperature found for all the implant conditions.

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